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Tennessee Agricultural and Industrial State University

Contract Numbers: N9onr-95700
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NR 055-214

"Preparation and Properties of Organic
Derivatives of Tin Hydride"

Final Report

May 31, 1953

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J. R. Lawson, D. E. Simmons, and J. B. Yeldell

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I. INTRODUCTION

This final report summarizes the research done at Tennessee Agricultural and Industrial State University under sponsorship of the Office of Naval Research according to the terms of contract N-9 onr 95700 and amendments. The goals of this project were:

- (1) to prepare and study the properties of a number of organic derivatives of tin hydride, (stannane).
- (2) to deduce from the differences between the properties of these compounds and those of analogous compounds of carbon and silicon; the effects due to the more electropositive nature of tin.
- (3) to derive, wherever possible, theoretical principles which will serve as a guide to further research in this area.

The choice of problems has been dictated by the availability of apparatus and personnel. A literature survey revealed that although considerable research on organo-tin compounds has been reported, comparatively little data of use to this investigation existed. Consequently it has been necessary to determine physical constants of already known compounds, as well as those of new compounds.

Details of the accomplishments made during the first three contract periods were given in a technical report dated August 31, 1952. The new work reported herein is an extension of the earlier investigations. For brevity, references to the previous technical report will be denoted by the symbol TR-1.

It should be noted that several aspects of this investigation may be considered still incomplete. This is to be expected because the results obtained thus far have logically suggested new problems. It is hoped that some of these problems can be attacked under another contract.

II. THERMOCHEMICAL STUDIES

It has been shown that the isobaric heat of combustion at 27°C of symmetrically substituted alkyl stannanes increases in a regular manner by about 157 kcal per mole per CH₂ group increase in the alkyl radical chain. (TR-1, page 10) The heats of combustion, $-\Delta H_{300}$, of tetra-alkyl tin compounds other than tetramethyl tin may be represented by the equation

$$-\Delta H_{300} = 157n + 292 \quad (1)$$

where n is the number of carbon atoms in the molecule. Other empirical equations relating the heat of combustion to the number of -CH₂ groups in the molecule also fit the data but no significant advantage over equation (1) is realized. In order to further test the validity of equation (1), the heat of combustion of an unsymmetrically substituted compound, diethyldibutyltin was determined. A detailed description of the experimental techniques was given in the previous technical report TR-1, pages 3 to 6. The heat of combustion of this compound was found to be 2169 kcal per mole while the value calculated by equation (1) is 2176 kcal per mole, in reasonable agreement with the experimental value. These data are summarized in Table I.

In an attempt to ascertain why the heat of combustion of tetra-

methyltin does not fit the equation (1); the heats of combustion of trimethylethyltin, dimethyldioethyltin and methyltriethyltin were determined. The results are summarized in Table I where the heats of combustion of tetramethyltin and tetraethyltin are also included for comparison. It is seen in Table I that there is the expected increase in heat of combustion in progressively adding CH_2 groups going from tetramethyltin to tetraethyltin. However the increment is not a regular one. This irregularity is also shown in the values obtained for the heats of formation of these compounds. The wide variance indicates considerable error in the data despite the apparent precision of less than one per cent agreement in the values obtained for the individual compounds. Our current belief is that the departure from equation (1) is only apparent and due to experimental difficulties. Although in every experiment the combustion reaction appeared to be complete, it is suspected that an undetermined loss of sample occurred in transferring the easily volatile methyl-substituted stannanes from the weighing pipette to the bomb.

III. BOND ENERGIES

Using data obtained in this investigation and auxiliary data of Linus Pauling,⁽¹⁾ tin-carbon "bond energies" were estimated to

(1) L. Pauling, "Nature of the Chemical Bond" Cornell Univ. Press, Ithaca, 1940 pp. 52-58.

be 33 kcal per mole and 35 kcal per mole in tetramethyltin and tetraethyltin respectively. (TR-1 page 12).

IV. PREPARATION OF ORGANIC DERIVATIVES OF STANNANE

The preparation and purification of several tetraalkyl stannanes, alkyl tin halides and alkyl tin hydrides were described in TR-1. In

TABLE I
Heats of Combustion and Formation
of Tetra-alkyl tin Compounds

Compound	Observed - ΔH (comb.) Kcal/mole	Heat of Formation Kcal/mole	Calculated ΔH (comb.) (Eqn. 1)
Diethyldibutyltin	2,170 2,167 2,174 2,169 Mean 2,170	- 54	2176
Tetramethyltin*	934	- 9	920
Trimethylethyltin	1,031 1,042 1,029 Mean 1,034	- 53	1077
Dimethyldiethyltin	1,221 1,214 1,229 Mean 1,221	- 28	1234
Methyltriethyltin	1,359 1,357 Mean 1,358	- 54	1391
Tetraethyltin*	1,547	- 27	1548

*Previously reported in TR-1, page 7.

addition, the following mixed alkyl stannanes have been prepared: diethyldibutyltin, trimethylethyltin, dimethyldiethyltin and methyltriethyltin. Of these, diethyldibutyltin is reported for the first time and while the latter three compounds are known, ⁽²⁾ we have de-

-
- (2) E. Krause and A. von Grosse "Die Chemie der metallorganischen Verbindungen", photolith reproduction, Edwards Bros. Inc. Ann Arbor, 1943 pages 317, 318.
-

termined several physical properties not previously reported by other investigators. Among these properties were the densities and refractive indices, from which were calculated the molar refractivities. The calculated values thus obtained were then compared with the theoretical molar refractivities as calculated from bond refraction data. ^(3, 4) The individual compounds are described below.

-
- (3) K. G. Denbigh, Trans. Faraday Soc. 36 936, (1940).

- (4) R. West and E. G. Rochow, J. Am. Chem. Soc. 74, 2490 (1952).
-

Diethyldibutyltin was prepared by reacting an ether solution of diethyltin dichloride with an excess of butylmagnesium bromide. Purification of the crude product was achieved using the procedure described in TR-1, page 2.

Physical properties of the pure compound were as follows: boiling point, 112°C at 10 mm Hg; n_D^{20} , 1.4734; density, 1.1035 g/ml at 20°C; molar refractivity, found: 74.03 ml, theoretical, 73.7 ml. Analysis: found; Sn, 39.1%; C, 48.9%; H, 9.75%. Calculated for $C_{12}H_{28}Sn$; Sn, 40.8% C, 49.5%, H 9.7%.

Trimethylethyltin was prepared by the reaction of methylmagnesium bromide with ethyltin trichloride in ether solution. Physical constants of the pure compound agreed with those reported in

the literature.⁽²⁾ The refractive index was found to be 1.4529 at
(2) Krause and Grosso, loc. cit.

20°C. The density at 20°C was 1243g/ml and the molar refractivity was found to be 41.87 ml. in agreement with the calculated value of 41.27 ml.

Dimethyldiethyltin was prepared by the reaction of methylmagnesium bromide with diethyltin dichloride. The refractive index was found to be 1.4601 at 20°C. Using the density given in the literature, the molar refractivity was found to be 45.96 ml. in agreement with the theoretical value of 45.90 ml. Other physical properties were in agreement with literature values.

Methyltriethyltin was prepared by the reaction of methyl magnesium bromide with ethyltin trichloride. Neither the density nor the refractive index of this compound has been reported previously. In this investigation the density was found to be 1.2205 g/ml. and the refractive index was found to be 1.4665, both properties being determined at 20°C. The molar refractivity was calculated from these data and found to be 50.17 ml. as compared with a theoretical value of 50.53 ml.

Diethyl Stannane: The preparation and properties of diethyl stannane, $(C_2H_5)_2SnH_2$, were described in TR-1, page 22. However, the vapor pressures were determined over but a small range of pressures and temperature. A larger quantity of this substance has been prepared and the vapor pressures were measured over a larger range. The plot of $\log P$ versus $(1/T)$ was linear and fit the equation, $\log P = \frac{-1576}{T} + 7.09$. By extrapolation, the normal boiling point was calculated to be 99°C.

V. INFRARED STUDIES

An account of the preliminary studies of the infrared absorption spectra of several tetraalkyl tin compounds was given in TR-1, pages 13-17.

Subsequently, the measurements made in the wavelength region of 2 to 16 microns have been checked and the study has been extended to include the wavelength region of 12 to 25 microns. Raman spectrograms of tetramethyltin and tetraethyltin have also been obtained. Details of these recent investigations are given below.

Experimental

The compounds were prepared in a state of high purity as was described in another section of this report. Spectrograms in the wavelength region 2 to 16 microns were made on a Perkin-Elmer "Model 21" dual beam recording spectrometer, with rock salt optics and with cams linear in wave number. The samples were contained in cells having rock salt windows and potassium bromide windows. The liquid cells were 0.1 mm. and .025 mm. in thickness. Samples of vapor were run in a 10 cm. tube at pressures varying from 2 to 25 mm. Hg.

Spectrograms in the wavelength region 14 to 25 microns were made at Vanderbilt University on a Perkin-Elmer "Model 21" instrument, with KBr optics, and with cams linear in wavelength.

The Raman spectra were obtained using the Vanderbilt physics Department's model 612 Hilger Spectrograph and an irradiation unit consisting of eight single-jacketed General Electric type AH-2 mercury arcs arranged symmetrically about the vertical Raman tube. Eastman 103-J and 103-O plates were used, and the excitation was by means of the 4358A mercury line.

Results

Spectrograms of nine organotin compounds and some tentative vibrational assignments of six symmetrically substituted alkyl tin compounds were presented in TR-1 pages 13-17. It was pointed out in this preliminary report that (1) it is possible to uniquely characterize each of the compounds of the type, SnR_4 , by means of its infrared spectrum; (2) the vibrational frequencies associated with carbon-hydrogen bond stretching, $\nu(\text{C-H})$ and deformation $\delta(\text{C-H})$ remain invariant throughout the homologous series of molecules going from tetramethyltin to tetra-n-hexyltin; (3) no skeletal vibrations appeared in the spectral range 2-16 microns for these compounds.

This investigation has been extended along the following lines:

- (1) the infrared spectral range has been extended to 25 microns;
- (2) Raman spectrographs have been made of tetramethyl and tetraethyl tin in order to reveal possible vibrational modes which are inactive in the infrared;
- (3) the investigation has been limited to the series, $\text{R}_y\text{SnR}'_{(4-y)}$ where R is the methyl radical and R' is the ethyl radical and y is an integer which varies from 4 to zero.

The observed infrared and Raman frequencies are presented in tables II, III, IV, V, and VI. In order to obtain sharp definition of all of the bands the samples were run at various liquid thicknesses and in the vapor state at various pressures. The data in Tables II-VI is thus a compilation from many individual spectrograms. A rather arbitrary legend has been used to denote the relative intensities of the bands. Assuming a hypothetical liquid thickness of 0.1 mm the following symbols are used:

Table II

Observed Infrared and Raman frequencies (in cm^{-1}) of
Tetramethyltin, $(\text{CH}_3)_3\text{Sn}$

Raman Liquid	Infrared Liquid	Infrared Vapor	Raman Liquid	Infrared Liquid	Infrared Vapor
	3678mw			1087m	1097mw
	3601w			1039ms	1041vw
	3530w	3063s		767vs	777vs
2980vs	2970vs	2972vs		734vs	
2920vs	2901vs	2901vs		697vs	
2601m	2356m	2374mw		681s	
	1863w		531vs		
	1728ms	1738mw	504vs		
	1709ms	1720mw	467w		
	1619m		449w		
	1506s		427w		
	1455vs	1456m	409w		
	1390s	1394mw	165vs		
	1357ms	1357mw	92ms		
	1271mw	1275vw			
	1247mw	1255vw			
		1218s			
		1208vs			
1186vs	1198vs	1201s			
1144vw	1131m	1146m			

Table III
Observed Infrared frequencies (in cm^{-1})
of Trimethylethyltin, $(\text{CH}_3)_3\text{SnC}_2\text{H}_5$

Infrared Liquid	Vapor	Infrared Liquid	Vapor
2928vs	3013m	1263m	
2897vs	2922s	1233s	
2857vs		1199vs	
2716m			1197ms
2594vw		1190vs	
2550vw		1015vs	1010m
2350m	2361w	1007vs	
2138w		959s	
2040w		939vs	939mw
1971m		760vs	790vs
1882vw		712vs	763vs
1715ms	1718vw	633m	
1690m		515s	
1583w		493s	
1470vs	1461mw		
1426vs	1435mw		
1379vs	1384mw		

Table IV
Infrared frequencies (in cm^{-1})
of Dimethyldiethyltin $(\text{CH}_3)_2\text{Sn}(\text{C}_2\text{H}_5)_2$

Infrared Liquid	Vapor	Infrared Liquid	Vapor
	2926s	1426vs	1429vw
	2900s	1379vs	1382vw
	2857ms	1259m	
2848vs		1233s	
2705ms		1189vs	1197w
2580w		1022vs	1011mw
2335m		957vs	
2131mw		939vs	938vw
1703m		740vs	740m
1676m		698vs	
1467vs	1466vs	513vs	

Table V
Observed Infrared Frequencies (in cm^{-1})
of Methyltriethyltin, $\text{CH}_3\text{Sn}(\text{C}_2\text{H}_5)_3$

Infrared Liquid	Infrared Liquid
2856vs	1426vs
2801vs	1379vs
2715ms	1234vs
2680mw	1189vs
2594vw	1011vs
2550vw	957vs
2344w	728vs
2140mw	670vs
1707mw	663vs
1677mw	508vs
1470vs	

Table VI
Infrared and Raman Frequencies (in cm^{-1})
of Tetraethyltin $(\text{C}_2\text{H}_5)_4\text{Sn}$

Raman Liquid	Infrared Liquid	Raman Liquid	Infrared Liquid
	3139m		1268m
	2842vs	1201vs	
	2813vs		1191vs
	2723s	1146vs	
	2689m	1089ms	
	2600w		1012vs
	2553vw	988ms	
	2374vw		961vs
	2147m		946vs
	1896w		803w
	1697mw	780ms	
	1676mw		774w
	1610mw		680vs
	1474vs		667vs
	1427vs	508s	
	1382vs	490vs	

vs-75-100% absorption

s-50-75%

ms-40-50%

m-25-40%

mw-15-25%

w-10-15%

vw-less than 10%

The spectra of the series $R_4\text{SnR}'_{(4-y)}$ revealed few differences in the rock salt region with the exception of tetramethyltin and tetraethyltin. However the two strong absorption bands very near 1250 and 1200 cm^{-1} characteristic of a 2,2-dimethyl grouping, were present in the spectra of all the compounds. This assignment has been confirmed by Simpson and Sutherland.⁽³⁾

(3) Simpson and Sutherland, Proc. Roy. Soc. A, 199, 169 (1949).

The attempt to remove the degeneracies by use of the unsymmetrical series $R_y\text{SnR}'_{(4-y)}$ and treating the methyl and ethyl radicals as point masses was successful only in the case of trimethylethyltin. This is because deviations from a true pentatomic tetrahedral molecule are introduced by the interactions between the internal and twisting motions of the ethyl group with the motions of the $\text{Sn-C}_2\text{H}_5$ skeletal vibrations. Hence it is highly dubious that the simplifying assumption of regarding CH_3 groups as point masses may be extended to heavier groups such as C_2H_5 . Nevertheless the calculations made by Simpson and Sutherland (loc. cit.) for X_3YZ molecules where Z is a large mass, X a CH_3 group and Y a carbon atom may be extended to include the case where the central

atom Y is a tin atom. On the basis of this work and that of Duncan and Murray,⁽⁴⁾ and Murata, Okawara,⁽⁵⁾ and Watase⁽³⁾ who have

(4) Duncan and Murray, J. Chem. Phys. 2, 636 (1943)

(5) Murata, Okawara and Watase, Ibid 18, 1308 (1950)

made assignments for tetraethyl lead and tetraethyl silicon respectively, assignments of skeletal vibrational modes for tetramethyltin, trimethylethyltin and tetraethyltin have been made and are presented in Table VII.

It is seen in Tables II and VI that the Raman data confirms several of the frequencies observed in the infrared. In addition the Raman spectra reveal, in the case of tetramethyltin and tetraethyltin, the infrared inactive symmetrical modes (ν_1 at 504 cm^{-1} and 490 cm^{-1} respectively, and ν_2 at 92 cm^{-1} for tetramethyltin. In the case of trimethylethyltin, the degeneracy of mode ν_3 , is strikingly revealed by the replacement of a methyl group by the ethyl radical.

The results obtained thus far indicate no peculiarities in the spectra attributable to the presence of tin rather than carbon or silicon. However by extension of the infrared measurements to longer wavelengths, (CsBr region) and by refining the techniques for obtaining Raman spectra of small amounts of liquid samples, distinctions due to the increased mass and more electropositive nature of tin will be observed.

VI. SUMMARY AND EVALUATION

As a result of this investigation, the following achievements can be cited:

- (1) Several significant differences between the organic derivatives of stannane and those of carbon and silicon

Table VII
Assignment of Observed Frequencies (cm^{-1}) of Alkyl Stannanes

Molecule	Skeletal Vibrational Modes*					
X_4Y	ν_1	ν_2	ν_3		ν_4	
$X_3Y Z$	ν_1	ν_4	ν_3	ν_2	ν_5	ν_6
$(CH_3)_4Sn$	504	92	531		165 cm^{-1}	
$(CH_3)_3SnC_2H_5$	633	(-)	1199	1263	(-1)	(-)
$(C_2H_5)_4Sn$	490	(-)	508		(-)	

(-) indicates region not yet observed

* notation from Herzberg "Infrared and Raman Spectra of Polyatomic Molecules"

have become apparent. (IR-1 pages 23-24)

- (2) It has also been shown that the heats of combustion of any tetraalkyl stannane can be predicted by means of a simple relationship,

$$-\Delta H_{300} = 157n / 292 \quad (1)$$

- (3) A number of new compounds have been prepared and descriptions of their properties have been added to the chemical literature. In this connection, recent theories on bond refraction have been tested and verified. Also improved analytical techniques have been developed.
- (4) Infrared studies have revealed the extent to which such techniques can be used as an analytical tool for the identification of organo tin compounds. Also a theoretical treatment of the vibrational modes of this series of compounds has been begun. For a complete treatment, extension of the measurements into longer wavelengths is indicated.
- (5) A most valuable accomplishment of this project not previously mentioned is the training of research personnel which it has afforded. Six persons on the student level and two persons with Master's degrees have received valuable experience in the use of the special techniques required in this work. Such experience has qualified them to receive governmental appointments with the Tennessee Valley Authority, research fellowships with the Atomic Energy Commission, research positions on other sponsored projects and positions in education and industry.

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